

Final Summary Report

June 10, 1964 to June 10, 1965

DEVELOPMENT OF MATERIAL SPECIFICATIONS AND QUALIFICATIONS OF POLYMERIC MATERIALS FOR THE JPL SPACECRAFT MATERIALS GUIDEBOOK

Prepared for:

JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA
JPL COGNIZANT ENGINEER: HUGH MAXWELL

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STANFORD RESEARCH INSTITUTE

MENLO PARK, CALIFORNIA



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SUMMARY

Three classes of materials, epoxide adhesives, silicone rubbers, and fluorocarbon films, have been studied under thermal vacuum conditions at 150°C and 10^{-6} mm Hg to determine their outgassing properties in a simulated space environment. Detailed results of these studies have been presented in Special Reports I-III. This Final Summary Report reviews these studies and presents some general conclusions about the thermal vacuum behavior of these materials.

INTRODUCTION

The over-all objective of this program was to provide assistance to the JPL staff members in the development of specifications and procedures for polymeric spacecraft materials. This includes definitions of properties, tests, and environments which are sensitive and meaningful, and collection of pertinent property, environmental, and materials data for use in specifications. Of particular importance to this program are the outgassing characteristics of various polymeric materials under thermal vacuum conditions. The classes of materials to be examined were selected by the JPL cognizant engineers.

The classes of materials selected for study were: 1) the general purpose epoxide structural adhesives, specifically the Epon¹ adhesives; 2) the RTV silicone rubbers,² for use as adhesives and potting compounds; and 3) Teflon FEP³ and Tedlar³ fluorocarbon film material. Samples were prepared and cured using recommended procedures, and their weight loss characteristics at 150°C and 10⁻⁶ mm Hg were determined. This temperature (150°C) approximates the currently recommended sterilization temperature for spacecraft. The results of this study have been reported in detail in three Special Reports.⁴⁻⁶

¹Trademark for Shell Chemical Company's epoxide adhesives

²General Electric Company's trademark for Room-Temperature Vulcanizing Silicones

³Trademark of E.I. du Pont de Nemours, Inc.

⁴Development of Material Specifications and Qualifications of Polymeric Materials for the JPL Spacecraft Materials Guidebook, Special Report No. I. Epoxide Adhesives

⁵Ibid., Special Report No. II, RTV Silicone Potting Compounds and Adhesives

⁶Ibid., Special Report No. III, Polyfluorocarbon Films

EXPERIMENTAL

Materials: - The materials used in this study, Epon adhesives, RTV silicone rubbers, and fluorocarbon polymers, were supplied by Shell Chemical Company, General Electric Company, and E. I. du Pont de Nemours and Co., respectively. Detailed sample preparations have been described in the Special Reports and in general follow the material preparations recommended by the manufacturers. In some cases, where the outgassing characteristics were poor, modified preparation and curing steps were investigated at the request of JPL cognizant engineer. These are detailed in Special Reports No. I and II.

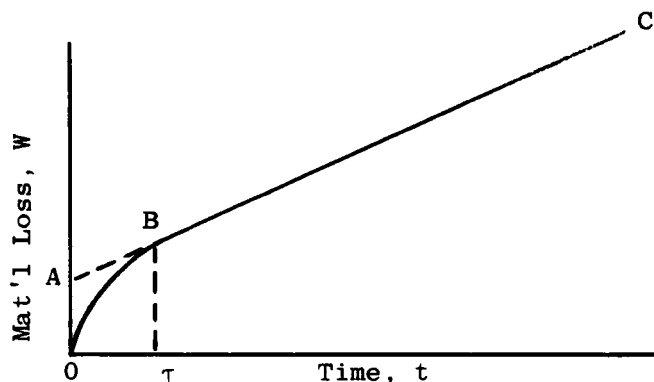
Apparatus: - The apparatus⁴ used in this study for thermal vacuum testing consisted of a high vacuum system with four sample cells heated to the desired temperature by oil baths. Pressures were measured by an ion gauge; the system was capable of routine pressures of 10^{-5} to 10^{-6} mm Hg.

Characterization of Materials: - Characterization and identification of the polymeric materials were performed using various spectroscopic methods. Infrared spectra were recorded on a Perkin-Elmer 221 Spectrophotometer, UV-visible spectra on a Cary Model 14, and mass spectra on a Consolidated Electrodynamics Corporation Model 21-103C.

Vacuum outgassing procedures were the same for all materials, i.e., the test specimens were subjected to thermal vacuum conditions of 150°C (in some cases 200°C) 10^{-6} mm Hg for 100-200 hours. The samples were removed at intervals to determine weight loss. Trapped outgassed volatiles were transferred from the cold trap into an evacuated glass bulb for mass spectroscopic analysis. In some cases, infrared spectra of condensable oils were obtained.

RESULTS

The outgassing curves of the materials tested took the form shown in the figure below, that is, there was a large initial weight loss, often taking place in the first 24 hours of thermal vacuum treatment; then the weight loss leveled off and varied linearly with time. Several useful parameters may be obtained from these curves; they are defined as follows:



Steady State is represented by the linear portion, (BC) of the curve.

Initial Weight Loss is the zero-time intercept of the linear portion of the weight loss curve (A).

Time until Steady State is the time required before the weight loss becomes linear (τ).

Steady State Loss Rate is given by the slope of the linear portion of the weight loss curve ($\Delta W/\Delta t$).

These parameters are extremely useful in summarizing the performance of a material under thermal vacuum conditions, and are included for all the materials tested at 150°C and 10^{-6} mm Hg. The data appear in Table I (Epon 924 and 941 are not included because they were eliminated on the basis of poor quality control); note, however, that this list includes only the materials prepared as recommended by the manufacturer. Special preparations or treatments of materials developed at SRI by the request of the JPL cognizant engineer are

described in the DISCUSSIONS Section. Table II summarizes the recommendations concerning the suitability for spacecraft applications of all the materials tested. A brief discussion of the results on each type of material is given below.

Epon Adhesives: - The nine Epon adhesives tested were classified according to their thermal vacuum performance at 200°C as well as at 150°C. The data on these materials are presented in detail⁴ in Special Report I. Epons 934, 931, 924, and 914 were classified as poor adhesives for spacecraft use because they undergo thermal decomposition between 150° and 200°C. Therefore, if use temperatures exceed 150°C, other materials should be used. Epons 941 and 903 are borderline materials; they degrade slightly at temperatures near 200°C but are quite stable at 150°C. Epons 901B-3 and 917 (with a slight modification of the curing cycle) have good outgassing characteristics even at 200°C, and are excellent adhesives for spacecraft use. This is also the case with Epon 422J if it is first postcured at 177°C to ensure complete cure and removal of the volatiles involved in the cure mechanism.

It has also been found that two factors are of considerable importance in determining the outgassing characteristics of these materials. The first factor is thickness. In testing a sample of Epon 914 at 150°C, any increase in thickness from approximately 0.4 to 4.0 mils resulted in a four-fold increase in percent weight loss. The second important factor in outgassing characteristics is material purity. In an experiment with Epoxide adhesive analogs, it was found that although the weight loss of the resin is fairly insensitive to the purity of the resin base, an impure curing agent greatly increases weight loss parameters.

RTV Silicone Adhesives and Potting Compounds: - The thermal vacuum testing⁵ of the RTV silicone compounds indicated that all were unsuitable for spacecraft use when cured as recommended by the manufacturer, owing to the large amounts of oil evolved under simulated space conditions. Attempts to eliminate this oil by postcuring in air at service temperature (150°C), or by increasing the amounts of curing agent formulated

into the resin were not successful since oil was still evolved from the test specimens. The oil was identified as low molecular weight silicone oils of the same chemical type as the base polymer. This oil can be removed from a silicone rubber by heating the rubber to 150°C under a vacuum of about 10^{-4} mm Hg for 24 hours; this treatment reduced greatly the amount of outgassed material from the silicone materials. It is realized, however, that this treatment may not be feasible for all spacecraft applications of the silicone rubbers.

Fluorocarbon Polymers: - All the polyfluorocarbon films tested in this study,⁶ including four types of Tedlar film and one Teflon FEP, had very low weight losses, all well below 1%, and, therefore, appear to be suitable for spacecraft use. The weight losses of the pigmented Tedlar films were, however, rather larger than those of the unpigmented transparent films (0.5% as compared with about 0.1%). There was also some slight discoloration of all the Tedlar films during thermal vacuum testing.

Table I

OUTGASSING PARAMETERS OF MATERIALS, PREPARED BY MANUFACTURERS RECOMMENDATIONS,
UNDER THERMAL VACUUM CONDITIONS OF 150°C and 10⁻⁶ mm Hg

Material	Time to Steady State	Initial Weight Loss, %	Wt. Loss rate %/100 hr	Wt. Loss after 200 hrs %	Initial Weight Loss g cm ⁻² x 10 ⁴	Wt. Loss rate g cm ⁻² hr ⁻¹ x 10 ⁶	Wt. Loss after 200 hrs g cm ⁻² x 10 ⁴
^a							
Epon Adhesives ^a							
Epon 903	< 24	0.78	0.10	0.98	1.6	0.21	2.0
Epon 914	< 24	0.49	0	0.49	4.6	0	4.6
Epon 901B-3	< 24	0.79	0.01	0.81	2.58	0.03	2.64
Epon 917	< 24	1.35	0.28	1.91	4.6	0.94	6.6
Epon 931	< 24	0.61	0	0.61	4.4	0	4.4
Epon 934	< 24	1.18	0.18	1.54	7.7	1.2	10.1
Epon 422J	< 24	3.97	0.23	4.43	17.1	1.28	19.7
^b							
RTV Silicones ^b							
RTV 511	75	4.33	0.21	4.75	107	5.2	117
RTV 560	35	3.36	0.10	3.56	93.4	2.7	98.8
RTV 11	55	2.72	0.18	3.08	72.5	4.8	82.1
RTV 615	70	1.93	0.18	2.29	44.8	4.2	53.2
RTV 60	55	1.62	0.06	1.74	49.0	1.7	52.4
RTV 112	100	6.07	0.07	6.21	14.0	0.1	14.2
RTV 108	100	5.90	0.11	6.12	11.8	0.1	12.0
RTV 106	100	5.42	0.09	5.60	7.4	0.3	8.0
RTV 102	100	5.95	0.05	6.05	16.8	0.1	17.0
^c							
Fluorocarbons ^c							
Tedlar 40/s	< 24	0.14	0	0.14	0.06	0	.062
Tedlar 30/A	< 24	0.39	.075	0.54	0.16	0.03	.22
Tedlar 30/B	< 24	0.35	.08	0.50	0.15	0.03	.22
Tedlar 20/s	< 24	0-0.05	--	0-0.05	0-.09	--	0-.09
Teflon FEP	< 24	0.08	< 0	0.08	0.10	0	0.10

^a Trademark for Shell Chemical Company Epoxide Adhesive

^b Trademark for General Electric Company Room Temperature Vulcanized Silicone

^c Trademark for E I du Pont de Nemours Polyvinyl Fluoride Film

Table II

CLASSIFICATION OF TESTED MATERIALS ACCORDING TO THEIR SUITABILITY
FOR SPACECRAFT APPLICATIONS^a

Material	Classification			Remarks
	Poor	Border-line	Satisfactory	
Epon 934	X	X	X	Degrades at < 200°C, high outgassing
Epon 903				Very high outgassing at 200°C, but not necessarily due to degradation
Epon 901B-3				Low outgassing at 150° and 200°C
Epon 917	X			High outgassing at 150°C
Epon 917 Longer cure ^b				Low outgassing at 150°C if cured 2 hrs at 177°C
Epon 931	X			Degrades at < 200°C
Epon 422J	X			High outgassing
Epon 422J Postcured				Very low outgassing at 150°C if postcured at least 6 hrs at 177°C
Epon 941	X			Cheaper product, poor quality control
Epon 924	X			High outgassing, poor quality control
RTV Silicones (potting cmpds & Adhesive/ Sealants)	X	X	X	Very high outgassing and evolution of condensable oils
RTV Silicones with Vacuum Postcure ^b				Low outgassing if postcured at 150° and 10 ⁻⁴ mm Hg for 24 hrs
All Tedlars			X	Very low outgassing
Teflon FEP			X	Very low outgassing

^a Materials prepared and cured using manufacturer's recommendation except where indicated

^b Materials cured using special treatments developed in the SRI program

DISCUSSION

In the thermal vacuum studies of polymeric materials, three parameters are especially useful in reporting data. These are the initial weight loss, the steady state weight loss rate, and the time required to reach steady state. The parameters were first defined by Fulk and Horr⁷ and have been quoted in Special Reports No. I-III. Of these, the initial weight loss is probably the least valuable in obtaining information on the long-term thermal vacuum stability of materials. This parameter reflects only the loss of surface contaminants and volatile species entrained within the polymer network. Of greater interest are the steady state loss rate and the time required to achieve steady state. It was found that when large amounts of relatively nonvolatile species were present, either as a result of degradation as in the case of some Epon adhesives, or as a result of additives as in the case of the RTV silicones, longer time is required to achieve steady state. When slow diffusion materials are not present, steady state is usually reached in less than 24 hours, since outgassed materials are usually air, water, etc. When slow diffusion materials are present, as much as 100 hours may be required to reach steady state. In materials where degradation is taking place, this time may be even longer or true steady state may not be achieved at all. High steady state loss rates seem to be indicative of material degradation. Thus the highest rates observed in this study were for the Epon adhesives such as Epon 934 which underwent degradation (with rates of the order of 1% per 100 hours) and the lowest, were for the very stable Teflon type polymers (approximately zero). Although the time required to attain steady state in the case of the silicone rubbers was large, rates were not very high, on the order of 0.1% per hundred hours, once steady state was reached.

⁷Fulk, M. M., and K. S. Horr, Trans. National Vacuum Symposium, 9, 324 (1962).

The constancy of weight loss rates has been assumed, but this is only an approximation. For example, Epon 917, which is considered a "good" spacecraft adhesive has a loss rate of 24.5×10^{-6} g per hour. Since the sample weight is only 0.8 g it would evaporate completely in about 5 years under these conditions, but it is obvious that the rate must be somewhat concentration-dependent, and the rate must decrease with time. The rate constants, however, provide a useful index for comparison of materials in the same time intervals (0-200 hours). To recapitulate: initial weight losses indicate the amount of volatile and surface contaminants; long times required in achieving steady state indicate the presence of high boiling contaminants which will condense readily and be quite troublesome in spacecraft; and a high, persistent weight loss rate may indicate actual material degradation.

Epoxide Adhesives:⁴ - Because these adhesives were tested at 200°C as well as at 150°C, much more insight was obtained into the thermal vacuum behavior of these materials. It was found that the 150°C data were somewhat misleading in that outgassing rates at this temperature did not reflect true thermal stability. Thus, although Epon 931 behaves as well at 150°C, having an initial weight loss of less than 1% and a zero rate, at 200°C it has a weight loss of well over 2% and a large loss rate. This material evolved degradation products in the form of condensable oils. The material, though apparently behaving well at 150°C, is unreliable: in any long exposures at 150°C, or if higher temperatures are reached, there is serious danger of degradation setting in.

On the other hand, Epon 422J behaved rather poorly at 150°C, having an initial weight loss of approximately 4%, but at 200°C the initial weight loss was only 4.4% and the loss rate had actually decreased. There was no evidence of material degradation, and the large initial outgassing was found to be due to inadequate curing rather than to material degradation. After a postcure was performed on this adhesive (six hours at 177°C in air) it was found that it appeared to be suitable for spacecraft use; i.e., initial weight losses were reduced to less than 1% at 150°C.

It may be concluded, therefore, that testing materials at only a single temperature is superficial at best and may result in erroneous conclusions concerning the suitability of a material. This is a particular problem for epoxide type adhesives, where the degradation temperature of the resin apparently depends more on the properties of the curing agent than on the resin base. Commercially supplied curing agents are often rather crude amine mixtures: the composition of these are extremely difficult to determine, and they are regarded as proprietary by the manufacturer. Since there are so many variations in epoxy formulation, it is advisable that the thermal properties of these materials be well known before they are recommended for spacecraft use.

The dramatic effect of curing agent purity was reported in Special Report I. The contrast between samples made up from pure m-phenylene diamine and those made up from practical grade (which is probably purer than that used commercially) is very striking. Initially each sample behaved rather similarly, but after treatment was well underway the samples containing pure amine leveled off somewhat while those containing the impure amine continued to show very large weight losses (2% after 100 hours compared to 1% after the same period for the pure amine).

Two types of modification are available for rendering materials suitable for spacecraft use; postcuring, or special treatment, of the cured material; and changes in formulation of the starting components. Postcures are suitable for materials like Epon 422J where the usual cure cycle is not adequate to bake out all volatiles in the sample. In a material like Epon 931 a basic change in adhesive formulation is needed--for example, use of a purer curing agent.

Silicones:⁵ - A rather different problem was encountered in the RTV silicone rubbers. Volatile condensable additives--low molecular weight silicone oils--are incorporated into the material by the manufacturer. It was found that these oils could not be bonded into the polymer network by increasing temperature of cure or catalyst concentration. The volatile oils could be removed only by subjecting the test specimens at 150°C and a vacuum of approximately 10^{-4} mm Hg for 24 hours. This

procedure yielded specimens having weight losses of less than 1%. It is realized, however, that this thermal vacuum postcure would be feasible in only a limited number of spacecraft applications. A change in material formulation to eliminate the volatile condensable component would seem the more desirable solution.

Polyfluorocarbons: - The fluorocarbon polymers tested presented few problems, and all seemed quite suitable for spacecraft use. All had initial weight losses of less than 1% and, with the exception of the pigmented Tedlar samples (types 30/A and 30/B), the weight loss rates of these materials were approximately zero. The weight loss rate of the pigmented samples was not large, but it might be of interest to see if a less volatile filler might not be more satisfactory.

CONCLUSIONS

It is realized that the data presented in Special Reports I-III, as well as in this final Summary Report, are not absolute but are a function of sample size, geometry, and of the method by which the test specimen is prepared. This makes apparent the need for rigorous criteria in determining outgassing parameters for specification purposes, so that the data obtained may be sensitive and meaningful.

The most striking feature of these studies, however, was the relative unimportance of the thermal stability of the base polymer in determining the outgassing properties of a given commercial material at 150°C. With the exception of a few epoxy resins, notably Epon 931 and 934, the primary reason for undesirably high outgassing was the presence of impurities or additives. In the case of epoxy adhesives, it was found that small amounts of impurity in the amine curing agent could have a profound effect on the weight loss of a resin as a whole. In the RTV silicones, low molecular weight silicone oils added by the manufacturer rendered the materials unsuitable for spacecraft applications

despite the fact that the silicone polymer was quite stable at 150°C. Even in the polyfluorocarbon films, which had extremely low outgassing properties, it was found that the weight losses of the pigmented films were several times greater than those of the unpigmented transparent materials.

Closely related to the impurity problem were the effects of inadequate curing conditions. For example, with Epon 422J, too brief a curing cycle failed to remove large amounts of volatile products resulting from the curing reaction. It is apparent, therefore, that a need exists for research on polymer materials, formulations, and preparative procedures in order to have material suitable for spacecraft use.

REFERENCES CONSULTED

The following references are not intended as a comprehensive bibliography on space-environmental effects of polymeric materials. However, these papers and reports were found to be useful on this project and would provide a general introduction to the problems encountered during tests of thermal vacuum effects on plastic materials, specifically the classes of materials studied here.

General

Air Force Materials Laboratory, Material Design Handbook, MLTDR-64-141.

Boebel, C. P., N. A. Mackie, and C. C. Quaintance, Outgassing Studies of Space Materials, Trans. Nat'l. Vac. Symp., 9, 307 (1962).

Broadway, N. J., R. W. King, and S. Palinchak, Space Environmental Effects on Materials and Components, Elastomeric and Plastic Materials, Vol. I., U.S. Dept. of Commerce AD 603364 (April, 1964).

Broadway, et al., Ibid, Appendix A-Elastomers.

Broadway, et al., Ibid, Appendix B-Plastics.

Broadway, et al., Ibid, Appendix E-Electrical Insulation.

Broadway, et al., Ibid, Appendix F-Laminates.

Broadway, et al., Ibid, Appendix G-Potting Compounds.

deWitt, E. A., et al., Effect of Low Pressure at Elevated Temperature on Space Vehicle Materials, Martin (Baltimore) Research Memorandum RH-29 (March 1959).

Fulk, M. M. and K. S. Horr, Sublimation of Some Polymeric Materials in Vacuum, Trans. Nat'l. Vac. Symp., 9, 324 (1962).

Gloria, H. R., W. J. Stewart, and R. C. Savin, Initial Weight Loss of Plastics in a Vacuum at Temperatures from 80° to 500°F., NASA TN D-1329.

Hamman, D. J. and E. M. Wyler, Space Environmental Effects on Materials and Components, Volume II. Electronic and Mechanical Components, AD601876 (April, 1964).

Jaffe, L. D. and J. B. Rittenhouse, Behavior of Materials in Space Environments, J.P.L. TN 32-150 (November, 1961).

Jaffe, L. D., Effects of Space Environment Upon Plastics and Elastomers, Chem. Eng. Prog. Symp., 40, 59, p. 81.

Levi, D. W., Literature Survey on Thermal Degradation, Thermal Oxidation and Thermal Analysis of High Polymers, for Plastics Technical Evaluation Service, (Picatinny Arsenal), June, 1963.

Madorsky, S. L. and S. Straus, Thermal Degradation of Polymers at Temperature up to 120°C. WADC TR 59 64 (March, 1960).

Parker, J. A., H. R. Gloria, and J. J. Lohr, Some Effects of the Space Environment on the Physical Integrity of Plastics, AIAA Annual Structures and Materials Conference, Fifth, Palm Springs, Calif., April 1-3, 1964.

Podlaseck, S. and J. Suhorsky, Stability of Organic Materials in a Vacuum, AD 453 239 (April, 1963).

Podlaseck, S., J. Suhorsky, and A. Fisher, The Behavior of Organic Materials at Elevated Temperatures in Vacuum, Trans. Nat'l. Vac. Symp., 9, (1962).

Rivera, M., et al., The Low-Pressure Gas Desorption of some Polymeric Materials, Trans. Nat'l. Vac. Symp., 9, 342 (1962).

Ringwood, A. F., Behavior of Plastics in Space Environments, Modern Plastics p. 173, Jan. 1964.

Riehl, W. A., Considerations on the Evaporation of Materials in a Vacuum, Chem. Eng. Prog. Symp., 40, 59, p. 81.

Epoxide Adhesives

Madorsky, S. L. and S. Straus, Stability of Thermoset Plastics at High Temperatures, Modern Plastics, p. 134, Feb. 1961.

Neiman, M. B., et al., Thermal Degradation of Some Epoxy Resins, J. Polymer Sci. 56, 383 (1962).

Anderson, H. C., Thermal Degradation of Epoxide Polymers, J. Applied Polymer Sci., 6, 22, 481 (1962).

Silicone Rubbers

Barry, A. J. and H. N. Beck, Silicone Polymers, Inorganic Polymers (F. G. A. Stone and W. A. G. Graham, ed.), Academic Press, New York, 1962, p. 189-307.

Hughes, J. S., "Some Recent Advances in Silicone Chemistry", Development in Inorganic Polymer Chemistry (M. F. Lappert and G. J. Leigh, ed.) Elsevier Pub. Co., Amsterdam, 1962, p. 138-161.

Fluorocarbons

Florin, R. E., L. A. Wall, D. W. Brown, L. A. Hymo, and J. D. Michaelson, Factors Affecting the Thermal Stability of Polytetrafluoroethylene, J. Res. Nat'l. Bur. Std., 53, 121-30, (1954).

Madorsky, S. L., V. E. Hart, S. Straus, and V. A. Sedlak, Thermal Degradation of Tetrafluoroethylene and Hydrofluoroethylene Polymers in Vacuum, J. Res. Nat'l. Bur. Std., 51, 327-333 (1953).

Miltek, J. T., A Survey Materials Report on Tetrafluoroethylene (TFE) Plastics, U S. Dept. of Commerce AD 607798 (Sept. 1964).